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# FIB/SEM Tomography of Porous Ceramics

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Porous ceramics have many important industrial applications among which are refractory ceramics for glass melting and honeycomb ceramic substrates for environmental applications. Controlling porosity, surface area, permeability etc. of these materials is critical for desired quality and functionality of the products based on or manufactured using these materials.

3D visualization enabled by dual beam microscopes combining Focused Ion Beam and Scanning Electron Microscopy (FIB/SEM) technology became a versatile materials morphology characterization technique [1-5]. It provides higher voxel resolution compared to X-ray tomography – tens of nanometers or, in some cases, even several nanometers. Even higher resolution is achievable by Transmission Electron Microscopy (TEM) tomography. However, it limits the analysis to a much smaller domain. Further sophistication of analysis is achieved by collecting EDX signal along with the secondary electron (SE) or backscattered electron (BSE) signal, albeit with a reduced resolution and significantly increased time consumption.

Post-experimental processing of the FIB/SEM tomography data, which is based on the image contrast, can be followed by the geometrical or statistical analysis of the structural components of the sampled volume. In particular, porosity, surface area and pore size distribution (PSD) can be computed. Another layer of analysis that relates morphology to the materials properties is provided by the application of finite volume or finite element methods to the obtained 3D structures. As a result absolute permeability and tortuosity values can be extracted (among other properties). Moreover, gas or liquid flow through the sample can be simulated with velocity and pressure field outputs inside the pores for given boundary conditions. Figure 1 shows an experimental FIB/SEM tomography slice of a sample prepared by embedding silica beads in epoxy (FIG. 1 (a)); the reconstructed 3D structure of the silica phase (FIG. 1 (b)); air velocity field in a plane perpendicular to the inlet direction, computed for a 0.03 m/s inlet velocity and output atmospheric pressure boundary conditions (FIG. 1 (c)).

There are several factors that limit either the quality of the experimental FIB/SEM tomography data or the thresholding of different phases or pores in ceramics. This study highlights practical aspects for obtaining good experimental data, imaging artifacts, as well as post processing and 3D reconstruction. Further, it reports on different examples of FIB/SEM tomography of materials based on SE and EDX signals. This includes a test sample prepared by embedding silica beads in epoxy, a zirconia silicate refractory and a ceramic substrate for exhaust filters. Porosity, PSD, absolute permeability and tortuosity computations based on a finite volume method are underway. Studies of silicate glass cracking due to nano-indenting by FIB/SEM tomography are envisaged.

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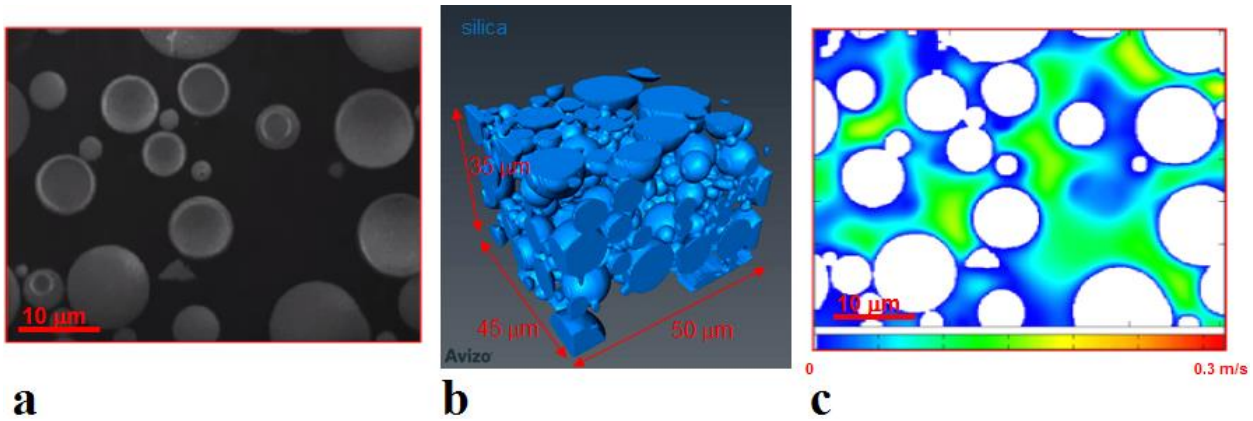


**Figure 1.** (a) FIB/SEM SE tomography slice of silica spheres embedded in epoxy, taken at 5kV, using FEI Quanta 3D FEG 600 instrument. (b) 3D structure of the silica phase reconstructed and visualized using Avizo® 9.0 3D software. (c) Air velocity field in a plane perpendicular to the inlet direction, computed for a 0.03 m/s inlet velocity and output atmospheric pressure boundary conditions, using TOMA software based on finite volume simulation code.

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